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S. W. STRATTON, DIRECTOR

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## EXTRACTION OF RUBBER GOODS

BY

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*Bureau of Standards*

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# EXTRACTION OF RUBBER GOODS<sup>1</sup>

By S. W. Epstein and B. L. Gonyo

## CONTENTS

	Page
1. Introduction.....	3
2. Preliminary experiments.....	3
3. Discussion of preliminary results.....	8
4. Effect of mixed solvent on free sulphur.....	10
5. Extraction of high-grade stocks.....	11
6. Effect of solvents on rubber.....	11
7. Summary.....	13

## 1. INTRODUCTION

In rubber analysis, where rubber is determined indirectly as the difference between 100 per cent and the sum of the organic extracts, ash, and total sulphur, it is important that the extracts represent as much as possible of the organic materials which were added to the rubber batch and which are not determined separately. Those who have tried to extract glue from a vulcanized compound containing it, by means of boiling water, know how tenaciously rubber clings to certain admixed materials. In this work it was our intention to ascertain as far as possible how much, if any, soluble organic material is retained in a compound after it has been extracted with acetone for eight hours and chloroform for four hours in the usual way.

## 2. PRELIMINARY EXPERIMENTS

For this purpose we chose a cheap rubber mixture containing mineral rubber and pine tar and extracted it successively as follows:

### Experiment I

Extracts	Hours	Per cent
(a) Acetone extract.....	8	10.56
(b) Chloroform extract.....	4	2.00
(c) Mixture of chloroform and acetone.....	4	2.00
Total.....		14.56

<sup>1</sup> Paper read before the rubber division of the American Chemical Society at its Philadelphia meeting, September, 1919.



## Experiment II

Extracts	Hours	Per cent
(a) Acetone extract.....	8	10.16
(b) Chloroform extract.....	4	2.0
(d) Mixture of carbon bisulphide and acetone.....	4	3.5
Total.....		15.66

Extracts (c) and (d), 2.0 and 3.5 per cent, respectively, are not removed in the standard procedure and therefore give us the first indication that organic material is retained in the rubber after the usual extractions have been carried out.

The residues from Experiments I and II were now digested in 25 cc of cresol (B. P. 195°) for four hours at 165° C. The solution obtained was then diluted with petroleum ether and allowed to stand over night. In the morning it was filtered upon a Gooch crucible and washed thoroughly with petroleum ether and boiling benzene. The filtrate contained vulcanized rubber, while the pad presumably retained only the fillers. Our experience with cresol as a solvent for vulcanized rubber has shown that there is on the pad no material soluble in acetone or chloroform when a compound containing only rubber, sulphur, and inorganic filler is treated with cresol in the manner given above. However, when this compound, which contained pine tar and mineral rubber, was treated in the same way, acetone extracted (I) 1.6 per cent and (II) 1.12 per cent, respectively, from the material on the pad. These figures represent a part of these organic additions which remained in the rubber after it was extracted.

This extract seems to have been released by the dissolution of the rubber in the compound. Further, when the pad was washed with hot 10 per cent hydrochloric acid and water, and then extracted with acetone again, the following extracts were obtained: (I) 3.28 per cent and (II) 3.88 per cent. In this case this material was retained by the fillers themselves and was released when they were dissolved out. A mixture of 50 per cent acetone and 50 per cent carbon bisulphide extracted (I) 1.64 per cent, and (II) 0.92 per cent additional

Arranging the extracts in the order in which they were obtained, our results are:

## Experiment I

Extracts	Per cent
Acetone extract on original sample.....	10.56
Chloroform extract on residue.....	2.0A
Additional extract obtained by means of a mixture of chloroform and acetone.....	2.0B
Acetone extract on the total fillers which were separated from the rubber by means of cresol....	1.6
Acetone extract on the residue after it had been washed with 10 per cent hydrochloric acid.....	3.3C
Additional extract obtained by washing with a mixture of carbon bisulphide and acetone.....	1.6
Total extracts.....	21.1

Of this total of 21.1 per cent of extractable material only 12.56 per cent is obtained in the regular procedure, while B and C, totaling 8.5 per cent, are left behind and are therefore reported as rubber hydrocarbon.

## Experiment II

Extracts	Per cent
Acetone extract on the original sample.....	10.16
Chloroform extract on the residue.....	2.00
Additional extract obtained by means of a mixture of carbon bisulphide and acetone.....	3.5
Acetone extract on the total fillers which were separated from the rubber by means of cresol....	1.12
Acetone extract on the residue after it had been washed with 10 per cent hydrochloric acid.....	3.88
Additional extract obtained by washing with a mixture of carbon bisulphide and acetone.....	.92
Total extracts.....	21.58

In this case, of the total of 21.6 per cent of extractable material only 12.2 per cent is obtained in the regular procedure, while B and C, totaling 9.4 per cent, are left behind.

It is easy to see, therefore, that in the case of cheap compounds the figure for rubber obtained by the "difference" method can not be at all reliable because of the retention of the organic material extracted as under B and C.

As long as the ashing method is used, it is clear that an accurate figure for rubber can not be expected for this reason. At best it can be but an approximation.

In connection with this work attention is drawn to the use of mixed solvents, such as acetone-chloroform and acetone-carbon bisulphide, which were used above. It was found with Experiment I that a mixture of chloroform and acetone extracted 2 per cent in four hours after the usual acetone and chloroform extractions had been run. Likewise, with Experiment II a mixture of carbon bisulphide and acetone extracted 3.5 per cent in the same

way. Such additional extracts, although they do not remove all of the material, nevertheless make the approximation closer. Considerable work has been done on the effectiveness of such mixed solvents.

When washing the pad through which a cresol solution of rubber had been filtered, it was discovered that usually a second extract of considerable weight could be obtained by subjecting the pad to alternate washings with acetone and carbon bisulphide after it was presumably free from all extractable material. We found that the coloration due to material being extracted out lasted only a short time, but reappeared on changing to the other solvent. The effectiveness of such alternate washings we believed to have been the result of a mixture of the solvents which was formed on the pad by the last few drops of the one solvent with the first few of the second which was being poured on. To prove this we tried washing with a mixture of equal volumes of the two solvents. The solvent power of such a mixture we found to be surprising. After washing with this mixture it was found that no further extract could be obtained by the alternating method mentioned above. This result of washing alternately with two solvents was noticed when we were working on the determination of free carbon in rubber goods, but we did not at that time grasp its significance. In this determination the rubber sample is treated with hot nitric acid, this solution filtered upon a Gooch, and the pad washed with nitric acid, acetone, and chloroform. It was found that this same pad, presumably free from extractable material, gave further extracts in many cases when the solvents were alternated.

Finding that such mixtures as acetone-carbon bisulphide and acetone-chloroform were more effective than the simple solvents in washing residues on Gooch pads, it occurred to us that if a constant boiling mixture of these solvents could be obtained this mixture might find application in the extraction of rubber goods in the Soxhlet.

Ryland<sup>2</sup> found that a mixture of 55 per cent carbon bisulphide and 45 per cent acetone by volume has a constant boiling point of 39.25° C.; likewise a mixture of 68 per cent chloroform and 32 per cent acetone by volume boils constantly at 64.7° C. Accordingly it was decided to extract samples of cheap rubber with acetone for eight hours and then with such a constant boiling mixture, and compare the weight of extract so obtained with that obtained by means of pure carbon bisulphide or pure chloroform. The following is a record of our work:

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<sup>2</sup> Ryland, *Jour. Amer. Chem. Soc.*, 22, p. 384; 1899. See also Young, *Fractional Distillation*, pp. 68 and 69.



TABLE 1.—Extracts Obtained From Low-Grade Mixings

Sample	Acetone extract eight hours	Chloroform extract on acetone extracted sample four hours	Carbon bisulphide extract on acetone extracted sample four hours	55 per cent carbon bi- sulphide, 45 per cent acetone on acetone extracted sample four hours	68 per cent chloroform, 32 per cent acetone on acetone extracted sample four hours	Total	Sum of the extracts
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	
I	9.8	2.0	1.8			11.8	Acetone extract+chloroform extract
	9.9					11.7	Acetone extract+carbon-bisulphide extract
	9.8			6.4		16.2	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
II	10.3	3.4	1.9		6.8	16.6	Acetone extract+68 per cent chloroform-32 per cent acetone extract
	10.4					13.7	Acetone extract+chloroform extract
	10.3			4.5		14.9	Acetone extract+carbon-bisulphide extract
III	15.3	3.7	3.2		4.2	14.5	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	15.3					19.0	Acetone extract+68 per cent chloroform-32 per cent acetone extract
	15.3			6.5		18.5	Acetone extract+chloroform extract
IV	13.3	2.2	1.7		4.6	21.8	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	13.3					19.9	Acetone extract+68 per cent chloroform-32 per cent acetone extract
	13.3			4.3		15.0	Acetone extract+chloroform extract
V	13.3	.8			2.9	17.6	Acetone extract+carbon-bisulphide extract
	9.1		1.1			16.2	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	8.7			3.2		9.9	Acetone extract+68 per cent chloroform-32 per cent acetone extract
VI	9.2	.5			1.5	12.4	Acetone extract+chloroform extract
	13.7		.5			10.7	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	13.7			2.4		14.2	Acetone extract+68 per cent chloroform-32 per cent acetone extract
VII	13.7	.6			4.0	17.7	Acetone extract+carbon-bisulphide extract
	7.7		.4			16.1	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	7.7			2.5		8.3	Acetone extract+68 per cent chloroform-32 per cent acetone extract
VIII	7.7	.6				10.2	Acetone extract+chloroform extract
	4.7		.6			5.3	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	4.7			2.2		6.9	Acetone extract+68 per cent chloroform-32 per cent acetone extract
IX	12.6	1.5	1.2			14.1	Acetone extract+carbon-bisulphide extract
	12.7			1.8		13.9	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	13.0	.7				14.8	Acetone extract+68 per cent chloroform-32 per cent acetone extract
X	9.1		.5		.9	9.6	Acetone extract+carbon-bisulphide extract
	9.1					10.0	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract
	6.3	.6				6.9	Acetone extract+68 per cent chloroform-32 per cent acetone extract
XI	6.3		.5		1.1	6.8	Acetone extract+carbon-bisulphide extract
	6.3					7.4	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract

### 3. DISCUSSION OF PRELIMINARY RESULTS

It is plain from the figures tabulated here that chloroform is slightly better than carbon bisulphide as a solvent for extracting rubber; while the constant boiling mixture, whether it be 55 per cent carbon bisulphide and 45 per cent acetone, or 68 per cent chloroform and 32 per cent acetone, is certainly a much more efficient solvent than either carbon bisulphide or chloroform. These mixtures removed extracts that in some cases were astonishing. In Case I, carbon bisulphide extracted 1.82 per cent, while the mixtures extracted 6.4 and 6.8 per cent, respectively. Case VI is even more striking, for carbon bisulphide removed only 0.43 per cent, while the mixture of 55 per cent carbon bisulphide and 45 per cent acetone removed 2.54 per cent.

Further, these results indicate that one extraction with a constant boiling mixture may take the place of the two extractions, namely, acetone for eight hours followed by chloroform for four hours, according to common practice in the rubber laboratory to-day. The following work was carried out in order to ascertain whether such a substitution would be advisable. The mixture used in the work was made by distilling a mixture of 55 per cent by volume of carbon bisulphide and 45 per cent by volume of acetone over copper at a temperature of  $39.25^{\circ}\text{C}$ . The solvent therefore contained no sulphides or free sulphur, so that sulphur determined in the extracts could not be attributed to the solvent. Likewise, the mixture of 68 per cent by volume of chloroform and 32 per cent by volume of acetone was freshly distilled and contained no sulphur.

TABLE 2.—Extracts and Sulphur Obtained on Low-Grade Rubber Mixings

Sample	A (CH <sub>3</sub> ) <sub>2</sub> CO extract eight hours		B CHCl <sub>3</sub> extract after A four hours		C CS <sub>2</sub> - (CH <sub>3</sub> ) <sub>2</sub> CO extract after A four hours		D CS <sub>2</sub> - (CH <sub>3</sub> ) <sub>2</sub> CO on original sample eight hours		E CHCl <sub>3</sub> - (CH <sub>3</sub> ) <sub>2</sub> CO on original sample nine hours		F  Total		
	Ext.	S	Ext.	S	Ext.	S	Ext.	S	Ext.	S	Ext.	S	How obtained
XII	11.7	1.23	4.6								16.3		A + B
												1.23	Free S in A
	11.9				5.2						17.1		A + C
		1.04				0.07						1.11	Free S in A + C
XIII							16.9				16.9		D
								1.26				1.26	Free S in D
	10.9		2.5								13.4		A + B
		.82										.82	Free S in A
XIV	10.9				3.0						13.9		A + C
		.65				.16						.81	Free S in A + C
							15.4				15.4		D
								1.14				1.14	Free S in D
XV									16.7		16.7		E
										1.0		1.0	Free S in E
	9.5		2.1								11.6		A + B
		.8										.8	Free S in A
XVI	9.3				4.5						13.8		A + C
		.78				.04						.82	Free S in A + C
							13.2				13.2		D
								1.12				1.12	Free S in D
XVII									14.6		14.6		E
										.91		.91	Free S in E
	20.7		7.9								28.6		A + B
		.51		0.15								.66	Free S in A + B
XVIII	21.5				8.0						29.5		A + C
		.39				.06						.45	Free S in A + C
							29.4				29.4		D
								.60				.60	Free S in D
XIX									31.1		31.1		E
										.48		.48	Free S in E
	12.5		3.3								15.8		A + B
		2.15		.06								2.21	Free S in A + B
XX	12.4				3.5						15.9		A + C
		2.0				.07						2.07	Free S in A + C
							17.6				17.6		D
								2.09				2.09	Free S in D
XXI									18.6		18.6		E
										2.10		2.10	Free S in E
	20.5		7.2								27.7		A + B
		.53		.10								.63	Free S in A + B
XXII	20.4				7.8						28.2		A + C
		.58				.06						.64	Free S in A + C
							28.4				28.4		D
								.72				.72	Free S in D
XXIII	15.9		1.2								17.1		A + B
		1.74		.06								1.80	Free S in A + B
	15.7				1.6						17.3		A + C
		1.53				.05						1.58	Free S in A + C
XXIV							17.7				17.7		D
								2.15				2.15	Free S in D
	21.4		2.0								23.4		A + B
		.56		.08								.64	Free S in A + B
XXV	21.0				2.1						23.1		A + C
		.82				.06						.88	Free S in A + C
							24.1				24.1		D
								.91				.91	Free S in D



TABLE 3.—Comparison of Extracts Obtained by  $\text{CS}_2$ — $(\text{CH}_3)_2\text{CO}$ , in 12 Hours Extraction on Low-Grade Rubber Mixings with (A+B) and (A+C), Carried out as in Table 2

Sample	Ext.	S	Remarks
XX	20.0	.....	Acetone extract+chloroform extract—(A+B)
	.....	0.63	Free S in extracts
	21.2	.....	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract—(A+C)
	.....	.64	Free S in the extracts
	20.5	.....	Extract obtained by the use of 55 per cent carbon bisulphide-45 per cent acetone on original sample for 12 hours
XXI	.....	.77	Free S in this extract
	19.8	.....	Acetone extract +chloroform extract—(A+B)
	.....	.66	Free S in the extracts
	20.0	.....	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract—(A+C)
	.....	.77	Free S in the extracts
XXII	20.3	.....	Extract obtained by the use of 55 per cent carbon bisulphide-45 per cent acetone on original sample for 12 hours
	.....	1.07	Free S in this extract
	16.1	.....	Acetone extract+chloroform extract—(A+B)
	.....	.21	Free S in the acetone extract
	16.1	.....	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract—(A+C)
XXIII	.....	.28	Free S in the extracts
	16.7	.....	Extract obtained by use of 55 per cent carbon bisulphide-45 per cent acetone on original sample for 12 hours
	.....	.24	Free S in this extract
	13.8	.....	Acetone extract+chloroform extract—(A+B)
	.....	.26	Free S in the acetone extract
XXIV	14.2	.....	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract—(A+C)
	.....	.28	Free S in the extracts
	14.2	.....	Extract obtained by use of 55 per cent carbon bisulphide-45 per cent acetone on original sample for 12 hours
	.....	.26	Free S in this extract
	18.1	.....	Acetone extract+chloroform extract—(A+B)
XXV	.....	.12	Free S in the extracts
	17.5	.....	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract—(A+C)
	.....	.14	Free S in the extracts
	17.9	.....	Extract obtained by use of 55 per cent carbon bisulphide-45 per cent acetone on original sample for 12 hours
	.....	.14	Free sulphur in this extract
XXVI	9.8	.....	Acetone extract+chloroform extract—(A+B)
	.....	.10	Free S in the extracts
	9.6	.....	Acetone extract+55 per cent carbon bisulphide-45 per cent acetone extract—(A+C)
	.....	.12	Free S in the extracts
	9.5	.....	Extract obtained by use of 55 per cent carbon bisulphide-45 per cent acetone on original sample for 12 hours
XXVII	.....	.14	Free S in this extract

#### 4. EFFECT OF MIXED SOLVENT ON FREE SULPHUR

A study of the above results reveals: (1) That a small amount of sulphur, usually less than 0.1 per cent, is extracted by the chloroform; (2) that about the same amount of sulphur is extracted by the mixture of 55 per cent carbon bisulphide and 45 per cent acetone, when this mixture is used in place of chloroform; (3) that either mixture extracts from the original sample, in 8 hours, as much or more material than the sum of the extracts obtained by the 8-hour acetone extraction and the 4-hour chloroform extraction (samples XIII and XIV are notable examples of the greater extracts that are sometimes obtained by the use of



the mixed solvents); (4) that either mixture extracts from the original sample, in 8 hours, as much or more material than the sum of the extracts obtained by an 8-hour extraction with acetone and a 4-hour extraction with a mixture of 55 per cent carbon bisulphide and 45 per cent acetone; (5) that the free sulphur present in the extracts obtained by the use of the mixtures is usually from 0.1 to 0.3 per cent higher than the amount present in the acetone extract; (6) that there seems to be no advantage in extracting 12 hours or longer with the mixed solvents, since, as shown in Table 3, the results are relatively the same after 12 hours as after 8 hours; (7) that the mixture of carbon bisulphide and acetone does not decompose during the extraction to liberate sulphur or sulphur compounds and in this way affect the free sulphur determination; (8) that the mixture of 68 per cent chloroform and 32 per cent acetone produces heavier extracts than that of 55 per cent carbon bisulphide and 45 per cent acetone.

#### 5. EXTRACTION OF HIGH-GRADE STOCKS

The next step in the work was to determine the effect of the mixed solvents on high-grade stocks for the purpose of possibly being able to determine what varying solution effects they would have on vulcanized rubber. Four compounds containing A-70 per cent, B-85 per cent, C-95 per cent, and D-45 per cent, respectively, of rubber were extracted in eight hours with acetone and four hours with chloroform, and then one was subjected to four hours additional extraction with chloroform, another to four hours with 55 per cent carbon bisulphide and 45 per cent acetone, and a third to four hours with 68 per cent chloroform and 32 per cent acetone, with the following results:

Sam- ple	Process	A 70 per cent rubber	B 85 per cent rubber	C 95 per cent rubber	D 45 per cent rubber
1	Extract obtained by four hours additional extraction with chloroform.....	0.38	0.37	0.69	0.25
2	Extract obtained by four hours additional extraction with 68 per cent chloroform and 32 per cent acetone.....	.63	.69	1.23	.50
3	Extract obtained by four hours additional extraction with 55 per cent carbon bisulphide and 45 per cent acetone..	.05	.22	.21	.00

#### 6. EFFECT OF SOLVENTS ON RUBBER

All three solvents dissolve vulcanized rubber to an extent which seems to depend upon how heavily it is compounded. It is interesting to note that the mixture of 68 per cent chloroform and 32

per cent acetone exhibits this ability to dissolve rubber to a marked extent. The rubber dissolved by it in the above is nearly twice that dissolved by pure chloroform. On the other hand, the mixture of 55 per cent carbon bisulphide and 45 per cent acetone hardly exhibits this ability at all, so that the rubber dissolved by it is inappreciable as compared with that dissolved by chloroform or the mixture of chloroform and acetone. Obviously, this is a point in favor of the use of the mixture of 55 per cent carbon bisulphide and 45 per cent acetone. The point is well borne out when high-grade stocks are used to compare the extracts obtained by acetone, chloroform, and the mixture of carbon bisulphide and acetone.

Sample	Percentage of rubber	Combined extract obtained by extraction for eight hours with acetone and four hours with chloroform		Extract obtained by extraction for eight hours with a mixture of 55 per cent carbon bisulphide and 45 per cent acetone	
		Extract	Free sulphur	Extract	Free sulphur
E	45	2.40	0.72	2.0	0.71
F	45	2.7	.64	1.9	.63
G	75	4.5	.37	4.1	.38
H	45	2.7	1.7	1.7	1.8
I	85	4.3	.82	3.6	.76

The results obtained by the acetone-carbon bisulphide mixture on these high-grade compounds are in every case lower than the sum of the acetone and chloroform extracts, as would be expected from the work carried out on samples A, B, C, and D. The figures for free sulphur are also in accordance with previous statements given here.

It is evident, therefore, that it is highly advantageous to use the mixture of carbon bisulphide and acetone to take the place of acetone and chloroform, because of this less active solvent effect upon rubber, as shown.

Before recommending the adoption of this mixed solvent a few words must be said about deterioration of the solvent and blank extracts that will form in it. It was found that the freshly distilled mixture would give no blank and at the end of two weeks would still give no blank. However, at the end of a month's storage 75 cc of the mixture would give a residue which, although it contained no sulphur, yet weighed up to 0.006 g. Therefore it is advised that, where the mixture is being used in large quantities for routine analysis, it be redistilled over copper every week. Where it is used only occasionally it should be mixed up fresh and distilled before using.

## 7. SUMMARY

1. Extraction for 8 hours with acetone followed by 4 hours extraction with chloroform does not remove all soluble material from some rubber compounds.

2. After a rubber sample has been extracted with acetone it was found: (a) That chloroform in every case extracted slightly more material than carbon bisulphide; and (b) that constant boiling mixtures, such as 55 per cent carbon bisulphide and 45 per cent acetone, and 68 per cent chloroform and 32 per cent acetone, extracted from many cheap compounds considerably more material than either chloroform or carbon bisulphide.

3. The constant boiling mixture of 68 per cent chloroform and 32 per cent acetone exhibits a marked ability to dissolve vulcanized rubber, as contrasted to the mixture of 55 per cent carbon bisulphide and 45 per cent acetone, which exhibits this ability hardly at all.

4. It is recommended that the constant boiling mixture 55 per cent carbon bisulphide and 45 per cent acetone be used in place of acetone and chloroform to extract rubber samples since:

(a) It eliminates one extraction with the necessary weighings.

(b) Extraction is complete in 8 hours while the acetone and chloroform extractions require a total of 12 hours.

(c) The extraction of free sulphur is complete.

(d) A rubber analysis in which the mixed solvent is used is more accurate than that in which acetone and chloroform are used separately, because: (1) Little or no rubber is dissolved by this mixture, as compared with chloroform, which will in some cases dissolve considerable quantities; (2) the extraction of cheap rubber compounds is more complete, since the extracts obtained are greater than the sum of the acetone and chloroform extracts.

H. P. Roberts, L. S. Hanicker, assistant chemists, and W. E. Lange, laboratory assistant, Bureau of Standards, rendered efficient assistance in obtaining the data presented in Tables 2 and 3.

WASHINGTON, September 11, 1919.









